CHAPTER 14

Data Review Reports

14-1. Introduction.

Post digestion spikes are typically evaluated for trace metal analyses to assess the ability of a method to successfully recover target metals from an actual sample matrix *after* the digestion process has been performed. The PDS results are used with MS results to evaluate matrix interferences.

14-2. Criteria.

14-2.1. Frequency.

a. Like matrix spikes and matrix duplicates, the frequency of post digestion spikes is ultimately established from the project's data objectives. No PDSs may be required or a PDS may be required for every sample in the batch. In general, a PDS should *not* be required for a set of environmental samples when a *representative* MS sample is processed and the MS recovery is acceptable. Ideally, when a PDS is required, the matrix spike and PDS should be prepared from the same environmental sample.

> Note: Project documents (e.g., QAPPs) often require PDSs to be analyzed at the frequency specified in standard analytical methods (e.g., the CLP SOW requires a PDS for each sample). Unfortunately, the frequency for PDSs may be poorly or inappropriately defined some methods. For example, Method 6010B of SW-846 states that a PDS should be analyzed "whenever a new or unusual sample matrix is encountered." However, the term "new or unusual" is not well defined. Furthermore, even if "new or unusual matrix" were defined, PDSs would not be required to demonstrate performance if representative matrix spikes were processed and acceptable MS recoveries were obtained.

b. When project documents do not specify the PDS frequency or the PDS frequency is deemed to be inappropriate by the reviewer and an unacceptable matrix spike recovery is observed, use professional judgement to determine whether or not a PDS analysis should have been performed. For example, a PDS would not be required to confirm the presence of matrix interference if a serial dilution analysis (SDA) were performed and confirms the matrix effect. However, in the absence of a technically defensible rationale to do otherwise, assume that a post digestion spike (PDS) must be analyzed when the MS is unacceptable.

14-2.2. Acceptance Limits.

The acceptance range for each PDS recovery must be no wider than the corresponding acceptance range for the matrix spike recovery. When project-specific limits are not specified, an acceptance range of 85% to 115% is recommended when the concentration of the PDS is at least

two times the native sample concentration. An acceptance range of 80% to 120% is recommended when the spiking concentration is one to two times the native analyte concentration.

> Note: The acceptance range for the PDS specified in Method 6010B (75–125%) is wider than acceptance range for the MS (80–120%). When metal analyses are performed using Method 6010B and the spiking concentration is high relative to the native analyte concentration, the acceptance range for the PDS should be no wider than 80–120%.

14-3. Evaluation.

Examine the standard preparation logs to verify that the PDS contains all the target metals. Examine the sample preparation log to determine whether the PDS was prepared from the same sample used to prepare the MS. Review the Case Narrative and the PDS summary forms and note any PDS failures. Using the laboratory's PDS summary form, recalculate the PDS recovery for at least one target analyte and compare it to the reported value. The reported and calculated result must agree to within *two* significant figures.

14-4. Qualification.

a. PDS results are qualified using the same strategies for matrix spikes. In particular, compare the PDS spiking levels to the concentrations of the native analytes in the sample selected for spiking. If the native concentration of a target analyte in the sample (digestate) is high relative to the spiking concentration, then the PDS recovery may not be representative of actual method performance. Evaluate the PDS recovery when the spiking concentration is at least *two* times greater than the native analyte concentration (e.g., unless the spiking concentration is slightly less than two times the native analyte concentration and a gross failure occurs). If environmental samples were qualified (e.g., by the laboratory) for matrix interference but the spiking level for the PDS is low relative to the native analyte concentration, remove the data qualifiers.

b. If a single field sample is used to prepare the PDS and MS and the spike concentrations of both batch QC samples are at least two times greater than the native analyte concentrations, then evaluate the data as discussed below.

14-4.1. MS Recovery Acceptable and PDS Recovery Unacceptable.

If the MS (and LCS) recovery for a metal falls within the QC acceptance range but a PDS was analyzed and the PDS recovery is unacceptable, a matrix effect should *not* be suspected. The laboratory would normally be expected to reanalyze the PDS sample (digestate) to confirm the result. Contractual corrective action for unacceptable laboratory performance may be appropriate when a number of failures of this nature are observed and confirmatory reanalyses are not performed. When a problem of this nature occurs, it is recommended that the Project Manager be notified. It may be appropriate to request the raw data to perform a more comprehensive review. If there is a gross discrepancy between the PDS and MS recoveries for a particular metal (e.g., the MS recovery is within 80% - 120% but the PDS recovery is not within 50–150%), rejection of the data would constitute the most conservative approach.

14-4.2. MS Recovery Unacceptable and PDS Recovery Acceptable.

In general, if the MS recovery for a metal does not fall within the QC acceptance range but the PDS recovery is acceptable, then a matrix effect (associated with the preparatory process) should be suspected and the field sample results must be qualified on the basis of the matrix spike recovery. However, when historical data for the effect does not exist, the laboratory would normally be expected to perform a second digestion and reanalysis of the MS to confirm the result. The result would be confirmed if the MS recoveries and PDS recoveries for both sets of analyses were similar in magnitude and bias.

14-4.3. MS Recovery Unacceptable and PDS Recovery Unacceptable.

a. When both the MS recovery and PDS recovery for a particular metal fall outside of QC acceptance range in the same manner (i.e., the PDS and MS failures are of similar magnitude and the direction of bias is the same), confirmatory analyses are unnecessary. Assume that a matrix interference exists and use the most noncompliant recovery (the MS or PDS recovery) to qualify the data.

b. When both the MS and PDS are unacceptable, the laboratory should be expected to make a reasonable effort to correct for matrix interference before qualifying the field samples for matrix interference. Review the Case Narrative to determine what corrective actions were performed. Corrective actions for matrix interference may include the use of a different matrix modifier, different instrument operating conditions, the method of standard additions, internal standards, a different digestion or analytical procedure, and serial dilutions (if action levels can be met).

> Note: If project documents do not clearly demonstrate that the matrix spike sample is representative of the samples in the batch (which often occurs in environmental investigations), then the benefits of extensive corrective actions by the laboratory to minimize a matrix effect should be considered to be minimal. In other words, if the laboratory did not make a “reasonable” attempt to correct for the matrix interference, but the matrix spike sample is not representative of the samples in the batch, the lack of “representativeness” should be considered to be much more significant than the lack of corrective actions.

c. When the PDS and MS are prepared from two different environmental samples and the spike concentration is at least two times the native metal concentration, then evaluate the PDS as follows:

(1) If both the PDS and MS recoveries for a target metal fall outside of the QC acceptance range in the same manner (i.e., the PDS and MS failures are of similar magnitude and the direction of bias is the same), then assume matrix interference exists and qualify the data using the strategies discussed in Chapter 12.

Table 14-1
Evaluation of PDS and MS Data

%R [MS/PDS] [1]	Summary of Evaluation	References
PASS / PASS	Results not qualified.	
PASS / FAIL	1. Check for confirmatory analyses for the digestate 2. Request additional information from the laboratory and use professional judgement to either reject the data, qualify the data using the unacceptable PDS recoveries, or qualify the data using the acceptable MS recoveries.	Chapter 14-4.1
FAIL / PASS	1. Check for confirmatory analyses for the matrix spike. 2. Qualify for matrix interference based on the MS %R.	Chapter 14-4.2
FAIL / FAIL	1. Check if corrective action was taken to address the matrix interference. 2. Qualify field samples for matrix interference based upon the most noncompliant of the MS and PDS recoveries.	Chapter 14-4.3

Notes:1. It is assumed that the PDS and MS were prepared from the same environmental sample. A PDS or MS recovery is denoted to be in *FAIL* status when percent recovery, %R, does not fall within the recovery acceptance range.

(2) When the PDS recovery is acceptable but the MS recovery is not, use the MS recovery to qualify the associated field sample results. The laboratory may be required to analyze additional PDSs for the batch of samples (e.g., one PDS for every sample in the batch) when the matrix spike recovery is unacceptable. Under these circumstances, verify that the additional analyses were performed.

(3) When inconsistent PDS and MS recoveries are observed for two different samples in the preparation batch (e.g., the PDS recovery is biased high and the MS recovery is biased low), then the representativeness of the PDS and MS results for the remaining samples of batch must be carefully assessed. In particular, if the MS recovery for a soil sample is acceptable but the PDS recovery for a second soil sample is not, then the MS and PDS samples may not be representative of the remaining samples of the preparation batch and qualification of these samples may not be appropriate.